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A COMPARATIVE REVIEW OF CURRENT METHODS FOR THE DETERMINATION OF ORGANIC MATTER IN SEWAGE.

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DURING the past five years, largely through the efforts of the Committee on Standard Methods of the American Public Health Association,¹ methods for the determination of organic matter in sewage have been placed upon a more definite and uniform basis than was formerly the case. The Committee, while specifying certain methods as standard methods, are yet cognizant of the considerable variability in the practice in different places in this country, and the difficulties attending radical changes in existing methods in the case of the older and long-established laboratories.

With the view of bringing these different methods together in concise form, it is the purpose of the writers to compare briefly the current practices in use in the different laboratories in this country, in England, and in Germany. The discussion will be confined to the Kjeldahl process, the determination of nitrogen as free ammonia, oxygen consumed, and loss on ignition.

METHODS FOR THE DETERMINATION OF THE UNOXIDIZED NITROGEN.

The Kjeldahl process.—In the absence of a method whereby there may be obtained an accurate knowledge of the character and actual amount of the nitrogenous compounds which are normally present in sewage of domestic origin, the Kjeldahl process furnishes well-nigh indispensable information regarding the amount of nitrogen which occurs in sewage in complex atomic aggregations. This process, in use to some extent for a number of years, has finally become quite generally recognized as the most reliable means of determining the nitrogen of the organic matter, and as such has been recommended as a standard method for sewage work.

Standard American method.—The prescribed method for the determination of the total organic nitrogen in sewage embodies one or more of the salient features of the procedures used by Drown and Martin, Hazen,² and in England.³ In the standard

method, the nitrogen as free ammonia is first removed by distillation with steam, following which the sample is digested with sulphuric acid until colorless; a small crystal of potassium permanganate is added, the digestate made alkaline with an excess of sodium carbonate, and finally diluted to a volume of 500 c.c. A suitable volume of this solution is then distilled with steam, and the ammonia evolved determined in the usual manner. Catalytic agents are not used in this method, and no steps are taken for the preliminary reduction of nitrified nitrogen.

English methods.—The methods of analysis described by Fowler,³ by Rideal,⁴ and employed by McGowan⁵ may be taken as representative of English practices in sewage analysis.

McGowan and his associates have used the Kjeldahl process to a considerable extent to determine both the total unoxidized and organic nitrogen in sewage. Their methods include both distillation and a direct nesslerization of the neutralized digestate. Their practice in regard to the Kjeldahl process is essentially different from that in common use in this country, in that a reduction of nitrate nitrogen is considered necessary. This is effected by a very slow reduction with zinc and sulphuric acid, requiring five days for its completion. Their reports distinctly state that when the reduction is omitted, or when a shorter period than five days is employed, loss of nitrogen is inevitable.

A direct process described by them involves a neutralization of the digestate, and the addition of sodium oxalate for the removal of the calcium, following which a suitable portion of the settled solution is diluted and nesslerized. This method is not recommended by McGowan for general use, owing to the liability to turbid tubes, and to the necessity of preliminary reduction of nitrates before the actual Kjeldahl digestion is attempted. Rideal,⁴ however, describes a direct method of which he thinks well, without commenting upon the necessity for preliminary reduction of nitrate nitrogen. The tediousness of McGowan's method obviously detracts from its value for practical routine work.

German methods.—From the works of Farensteiner, Buttenburg, and Korn,⁷ and the reports of the Royal Bureau of Sewage Disposal at Berlin,⁸ we learn that the Kjeldahl process is there regarded as of great value in sewage work. The official method includes the destruction of all oxidized nitrogen by treatment with sodium sulphite, ferric chloride, and dilute sulphuric acid, after which follows the usual digestion, neutralization, and distillation. Owing to the high concentration of German sewages the final step is a distillation into standard acid rather than nesslerization. In some cases catalytic agents are used, such as platonic chloride and copper oxide,⁹ or copper sulphate or oxide.¹⁰ The German method of preliminary reduction is much more satisfactory than the tedious English method. Neither of these, however, is considered essential for American conditions.

Phelps' method.—In the work at the Sewage Experiment Station of the Massachusetts Institute of Technology, Phelps⁶ has used a Kjeldahl method wherein copper sulphate is employed as a catalyzing agent and permanganate is added at the completion of the digestion. Phelps shows very clearly the necessity for carrying the digestion to a point where a colorless solution will result, and brings forward data as to the feasibility of using permanganate; wherefrom he concludes that permanganate, although not essential in all cases, must still be considered a necessary adjunct to the reagents used in the Kjeldahl process as employed by Palmer.¹¹ He concludes his paper with suggestions in favor of the feasibility of determining organic nitrogen by direct nesslerization of the ammonia thus formed.

*Columbus method.*¹²—Following lines suggested by the English workers, a method for the direct nesslerization of the neutralized digestate of the Kjeldahl process was developed at the Columbus Sewage Testing Station. By applying the principles of water softening, there were eliminated the calcium and magnesium, whose interference has long been the cause of the turbidity besetting former attempts at direct nesslerization. The details of the method are given in another paper, but, in brief, the neutralized digestate is first treated with an excess of alkali, sodium carbonate is then added to precipitate the calcium, and under these conditions complete softening of the digestate results, allowing the clear yellow color of the mercury ammonium iodide to develop in a normal manner upon the subsequent nesslerization of a suitable portion of the clarified digestate. Potassium permanganate is omitted on the ground that manganous salts might escape oxidation during the preparatory treatment of the digestate, whereby turbid tubes would result on nesslerization, as experiments to cover this point have clearly indicated.

THE NECESSITY FOR PRELIMINARY REDUCTION OF NITRATES IN THE KJELDAHL PROCESS.

Some published literature is available on the effect of nitrate nitrogen upon the results of the Kjeldahl process. Rideal⁴ describes experiments wherein certain amounts of nitrate were added to sewages to the extent of 68 and 136 parts of nitrogen as nitrate per million, respectively. To these sewages the Kjeldahl process was applied, digesting with 2 c.c. and 4 c.c. sulphuric acid, respectively. The results of the experiment are shown in the following table, from which the conclusion was drawn that the large excess of sulphuric acid used in the Kjeldahl process prevents loss of nitrogen by secondary decomposition.

TABLE I.
DR. RIDEAL'S EXPERIMENTS AS TO THE EFFECT OF NITRATES UPON THE RESULTS OF THE KJELDAHL PROCESS.

C.C. SULPHURIC ACID ADDED	TOTAL NITROGEN BY KJELDAHL— PARTS PER MILLION		
	Parts per Million added Nitrate		
	0.0	68	136
2.....	425	350	375
4.....	410	375	390

NOTE.—Sample strongly urinous, contained 350 parts per million nitrogen as free ammonia.

Phelps, in a study of the Kjeldahl process, has published some convincing data with regard to the effect of nitrates. He added nitrate and nitrite nitrogen in varying amounts up to 40 parts nitrate

and 20 parts nitrite per million, respectively, to samples of crude sewage, septic sewage, coarse-grain filter effluents, and sand filter effluents, and determined the unoxidized nitrogen in the several samples before and after treatment. The results showed conclusively that no practical interference need be looked for in the examination by the Kjeldahl process of samples containing as high as 60 parts nitrified nitrogen per million.

The writers have also made experiments upon this point, determining by the direct Kjeldahl process the unoxidized nitrogen in samples of a settled sewage to which nitrate nitrogen was added up to 30 parts per million. From the results given below it is evident that the effect of nitrate is practically negligible.

TABLE 2.
EFFECT OF NITRATES UPON THE RESULTS OF THE KJELDAHL PROCESS.
(Average of Duplicate Determinations.)

PARTS PER MILLION NI- TRATE NITRO- GEN ADDED	NITROGEN—PARTS PER MILLION		
	Total	As Free Ammonia	Organic
0.....	15.4	6.4	9.0
10.....	15.4	6.4	9.0
20.....	15.0	6.4	8.6
30.....	14.6	6.4	8.2

NOTE.—0.1 difference on standard is equivalent to 0.4 part per million for 50 c.c. sample.

RELATIVE SUPERIORITY OF THE KJELDAHL PROCESS OVER THE ALBUMINOID AMMONIA PROCESS.

The albuminoid ammonia process has been in general use for many years in this country and in England, but only in recent years has been supplanted in the larger laboratories by the Kjeldahl process. The former process has, as we all know, long been recognized as yielding uncertain results, which represent only a portion of the total organic nitrogen obtainable by the use of the Kjeldahl process. The variability of the ratio between the albuminoid ammonia and the Kjeldahl values was well illustrated by the comparative serial analysis made during the staling of a sample of fresh sewage, as reported by Fuller.¹³ In this case the ratio varied from 20 per cent to 50 per cent, increasing as the septicity of the sewage increased.

The uncertainties of the albuminoid ammonia results have been shown by a number of observers.^{14 15 16 17 18 19 20}

TABLE 3.
COMPARATIVE DATA ILLUSTRATIVE OF THE VARIABILITY OF THE RELATION WHICH ALBUMINOID
NITROGEN RESULTS BEAR TO KJELDAHL NITROGEN.

CRUDE SEWAGE.				
LOCALITY	OBSERVER	NITROGEN PARTS PER MILLION		PERCENTAGE WHICH ALBU- MINOID NITROGEN IS OF NITROGEN BY THE KJELDAHL PROCESS
		As Albuminoid Ammonia	Kjeldahl	
Lawrence, Mass.....	Clark	3.0	7.8	39
Hopedale, Mass.....	"	2.2	6.0	37
Leicester, Mass.....	"	3.3	7.2	46
Manchester, Eng.....	Fowler	4.2	19.0	22
Manchester, Eng.....	"	4.8	15.5	31
Belfast, Eng.....	Letts	8.9	15.3	58
Saltley, Eng.....	Watson	15.6	39.1	40
Rhea, Eng.....	"	15.2	48.3	21
Hocklay, Eng.....	"	16.7	59.7	28
Aston, Eng.....	"	19.5	92.8	21
Columbus, Ohio*.....	Johnson and Kimberly	3.5	7.3	48

*Average of 22 analyses.

NOTES.—Massachusetts data compiled from Report Massachusetts State Board of Health, 1903. English data, from Manchester Reports and Royal Commission Reports, 1902, and Report of Birmingham, Rhea, and Tame Drainage Board, 1903

TABLE 4.
COMPARATIVE DATA ILLUSTRATIVE OF THE VARIABILITY OF THE RELATION WHICH ALBUMINOID
NITROGEN RESULTS BEAR TO KJELDAHL NITROGEN.

SEPTIC SEWAGE.				
LOCALITY	OBSERVER	NITROGEN. PARTS PER MILLION		PERCENTAGE WHICH ALBU- MINOID NITROGEN IS OF NITROGEN BY THE KJELDAHL PROCESS
		As Albuminoid Ammonia	Kjeldahl	
Lawrence, Mass.....	Clark	3.0	7.8	39
Hopedale, Mass.....	"	2.2	6.0	37
Leicester, Mass.....	"	3.3	7.2	46
Manchester, Eng.....	Fowler	4.2	19.0	22
Manchester, Eng.....	"	4.8	15.5	31
Columbus, Ohio.....	Johnson and Kimberly	3.6	6.4	56

SEWAGE SLUDGE.				
Brockton, Mass.....	Clark	56.0	181	31
Lawrence, Mass.....	"	13,000.0	23,700	55
Lawrence, Mass.....	"	390.0	860	45
Lawrence, Mass.....	"	633.0	1,109	56
Lawrence, Mass.....	"	731.0	1,644	44
Lawrence, Mass.....	"	222.0	294	76
Lawrence, Mass.....	"	530.0	943	56

EFFLUENTS OF COARSE-GRAIN SEWAGE FILTERS.				
Columbus, Ohio* }	Johnson and Kimberly	1.1	2.1	52
Columbus, Ohio† }		1.6	3.8	42

*Contact filter, average of 10 determinations.

†Sprinkling filter, average of 16 determinations.

From the Massachusetts and the English reports the tables on the preceding page have been prepared, which, while necessarily incomplete, may be said to illustrate the great variability of the ratio existing between the results by the two processes and the inherent cause of the decline of the albuminoid ammonia process.

METHODS FOR THE DETERMINATION OF NITROGEN AS FREE AMMONIA.

There are two methods for the determination of nitrogen in sewage in the form of ammonium salts, namely, by distillation with sodium carbonate or magnesia, or by direct nesslerization. The latter method is rapidly coming into general use. A digest of current thought and practice in this regard is as follows:

German method.—This method consists in a clarification of the sample with caustic soda. In certain cases zinc acetate is also added to remove hydrogen sulphide. Following this preparatory treatment, a suitable portion of the clear supernatant liquid is removed, diluted to 50 c.c., and nesslerized. Phelps²⁰ has modified the method by diluting the sample to double its original volume before adding the caustic soda, and by using copper sulphate and lead acetate as coagulants.

English method.—Rideal⁴ and McGowan⁵ describe a method for direct nesslerization. Rideal speaks highly of the process, while McGowan says it cannot be used in all cases, since turbid nesslerized tubes result under certain conditions, especially when hard effluents high in calcium are examined. There is an apparent lack of harmonious opinion in England regarding the feasibility of determining nitrogen as free ammonia by a direct process.

From our standpoint the cause of the complications referred to by McGowan are apparently due to the lack of sufficient carbonic acid ions to precipitate the incrusting calcium. In cases where a sewage high in sulphates is examined, in the absence of alkalinity, the technique should include the addition of sodium carbonates as well as caustic soda.

Columbus method.—An alternative method, applicable when the sewage contains high magnesium, consists in treating the sample with caustic soda in sufficient amount to precipitate the magnesium and to soften the sewage. A precipitate of magnesium hydrate and calcium carbonate results, whereby there is effected a complete clarification of the sewage. This modification is embodied in the Report of the Committee on Standard Methods.¹ For the typical hard sewage of Columbus, the presence of a lead salt was not required for the removal of sulphureted hydrogen. For sewages low in magnesium the method may be made applicable by the addition of a magnesium salt as a coagulant.

RELATIVE RESULTS BY THE DIRECT AND THE DISTILLATION METHODS.

The advantages attached to the direct methods of determining nitrogen in sewage in the form of ammonium salts refer to the fact that by distillation there is obtained a certain amount of ammonia, split off from partially reduced nitrogenous organic compounds on

the border line between ammonium salts and crude organic matter. Further, as has been shown by Phelps,²⁰ distillation with sodium carbonate causes the development of ammonia from a number of nitrogenous substances. Under such circumstances a direct process should give somewhat lower results, representative of true ammonium compounds alone. As pointed out by Schmidtman and Günther,⁸ the direct processes yield results now higher, now lower, than the distillation process, but generally, according to the experience of the writers, averaging about 90 per cent of those obtained by distillation. In the following table are shown the results obtained by the writers upon this point.

TABLE 5.
COMPARISON OF RESULTS BY THE DIRECT AND DISTILLATION METHODS FOR THE DETERMINATION OF NITROGEN AS FREE AMMONIA.

CHARACTER OF SAMPLES	NUMBER OF SAMPLES AVERAGED	NITROGEN AS FREE AMMONIA PARTS PER MILLION		PERCENTAGE WHICH DIRECT RESULTS WERE OF DISTILLED RESULTS
		Direct	Distilled	
Crude sewage.....	32	13.1	15.1	87
Settled sewage.....	33	13.2	14.8	90
Septic sewage.....	27	12.8	14.0	90
Strained sewage.....	13	14.2	15.9	90
Effluent of coarse-grain filter.....	39	10.6	11.8	90
Effluent of sand filter.....	49	2.23	2.5	90

THE DETERMINATION OF OXYGEN CONSUMED.

No method has undergone greater modification than that for the determination of the oxygen consumed by, or absorbed from, potassium permanganate.

Standard method.—Following the practice of Palmer,²¹ the Committee has recommended for use in newly established laboratories a 30 minute contact with acid permanganate in flasks immersed to the neck in a boiling water-bath. This period of contact and this technique were advised for the reason that a fairly complete oxidation is effected thereby, with a minimization of the personal errors incidental to the other modifications.

Lawrence method.—The practice at the Lawrence Experiment Station² is to boil for two minutes with an excess of permanganate, titrating back with oxalic acid.

Boston method.—This method²² is similar to the Lawrence method except that the period of boiling is five minutes. In both these methods the permanganate is added to the hot solution.

German method.—In Germany⁷ the period of boiling is uniformly 10 minutes, and the determination is usually made upon the *filtered* solution.

English methods.—In England the boiling methods are practiced but little, as considerable error is believed to be introduced through the decomposition of the per-

manganate itself at the temperature of boiling water. The English methods use the "thiosulphate-iodine" reaction for final titration, since oxalic acid is obviously unsuitable in the cold. The many modifications of the oxygen consumed test in current practice in England are as follows:

*The "at once" test.*²³—In this method, carried out at room temperature, the excess of permanganate is removed within 30 seconds after its application by the addition of potassium iodine.

The "three-minute" test.—In this method²⁴ the permanganate is allowed a contact of three minutes at a temperature of 80° F.

Other tests.—Periods of 15 minutes,²⁵ two and a half hours,⁴ and four hours, at 80° F.,³ are in use in England. Of these, the 15 minute and four hour periods are the more generally practiced. These modifications are said to serve the purpose of differentiating the putrescible matter. As stated by Rideal,⁴ at the Manchester Meeting of the Society of Chemical Industry in 1898, the several oxygen consumed tests were said to possess the following characteristics:

1. "The three minute test showed (nitrites, ferrous salts, sulphureted hydrogen and) putrefying matter decomposing permanganate at once with acid."
2. "The difference between three and 15 minutes showed matter readily putrefying and rapidly decomposing acid permanganate."
3. "The difference between 15 minutes and four hours gives matter capable of putrefying, though slow to decompose."

Rideal⁴ obtains what he calls a final figure by subjecting the sample to the action of permanganate for two and a half hours at a temperature of 80° C.

COMPARISON OF RESULTS BY DIFFERENT METHODS.

These different methods for determining the oxygen consumed or absorbed from acid permanganate bear a fairly definite relation to one another, as is illustrated by the following table:

TABLE 6.
COMPARISON OF RESULTS BY DIFFERENT METHODS.

SOURCE OF METHOD	TIME OF CONTACT (Minutes)	TEMPERATURE (°C.)	RELATIVE RESULTS TO 5 MINUTE BOIL—AUTHORITY		
			Columbus	Fuller	Kinnicutt
England.....	3	26.7	0.20	0.20
England.....	15	26.7	0.33	0.35	0.30
England.....	240	26.7	0.56	0.60	0.48
Palmer.....	30	96.0	3.14
"Absolute".....	240	100.0	4.73	4.00
Lawrence.....	2	100.0	0.74	0.65	0.65
Boston.....	5	100.0	1.00	1.00	1.00
Germany.....	10	100.0	1.18	1.25	1.52

LOSS ON IGNITION.

The ignition of the residue remaining upon evaporation of a measured portion of sewage has been practiced for a good many years. The loss in weight sustained is roughly indicative of the total

amount of organic matters present. As is well known, many other substances besides organic matter are volatilized during the ignition, so that the test does not strictly indicate the organic content of the sample. Notwithstanding the limitations of the method, the information afforded thereby is of considerable value in sewage work, as it is the only test whereby even an approximation of the total amount of organic matters present. Relative to questions of the disposal of sludge deposits, the data afforded have a particular field of usefulness.

Limitations of the process.—In brief terms, the loss in weight upon ignition includes all organic matters, a certain amount of carbonic acid and combined water split off from the carbonates of the alkaline earth metals, especially magnesium, and further, under certain conditions, a certain loss due to the deoxygenation of mineral salts in cases where the temperature of ignition has been too high. Chlorides are also a factor, when present in considerable amounts.

The factors of error have been corrected to a certain extent by the refinement of the technique of the method in its more recent development. At the same time, as is well known, the variable character of these complications cannot always be allowed for in an entirely satisfactory manner. Even under these conditions, the process is of considerable value, since it throws light upon those constituents regarding whose true nature, amount, and precise composition the present status of analytical methods yields but sparse information.

German method of determining loss on ignition.—During the tests made at Cologne, Germany, in 1904,⁸ loss on ignition data was obtained by a technique involving repeated partial ignitions at a very low temperature (moving the dish back and forth over a low flame), preceded by the application to the residue of distilled water and subsequent evaporation. From four to six ignitions were usually made; following the final heating, the residue was treated with a few drops of ammonium carbonate solution to convert to carbonate any alkaline earth oxides. With this technique, a very complete combustion of the organic matter was effected, with a minimum loss of volatile mineral matter. This method appears to be a distinct improvement over the technique employed in this country, and, as brought out in another paper,²⁶ the experience of the writers indicates that a

re-application of distilled water to ignitions made in the radiator following a second ignition might with advantage be included in the standard procedure, as the combustion of the organic matter would thereby be more complete and the results of the process more reliable.

Greater relative value of volatile suspended matter data.—Loss on ignition is of course not a test of differentiation. Although the data obtained by ignition of the residue from the evaporation of a measured amount of sewage are to be regarded only as approximations of the constituents they are intended to represent, it is possible, by the use of an entirely different process, to obtain precise information regarding that portion of the suspended matters in sewage which is of a volatile character. To effect this, the suspended matters are separated from the dissolved matters by filtration through asbestos in a Gooch crucible.²⁶ In the absence of the complicating mineral matters, the volatile suspended matters may be determined by an ignition at a temperature sufficiently high to effect a complete destruction of all organic matters. In problems relating to the preparatory treatment of sewage, these data are of great value, since they indicate to a certain degree the relative composition of the deposits of sludge which are incidental to these processes.

CONCLUSIONS.

Casting up the evidence, it seems apparent that more satisfactory practical results may be obtained by the Kjeldahl process than by the use of the albuminoid ammonia process, the results of which are such uncertain percentages of the total organic nitrogen present in sewage and in effluents.

The direct process for the estimation of the ammonia formed in the Kjeldahl method seems practical and reliable above 0.5 part per million organic nitrogen. Below this amount the distillation method of Palmer is available.

The direct determination of nitrogen as free ammonia is safe and accurate; either modification of the process may be used according to local hardness conditions.

Although governed largely by local conditions, the oxygen consumed test, with its many modifications, is to be considered of great value in the examination of crude and purified sewages.

Regarding the loss on ignition, the indications are that the total and volatile suspended matters, as determined directly by the Gooch process, are more reliable and more satisfactory data for sewage problems than either the total volatile matters in the sewage or effluent or the indirectly determined total and volatile suspended matters. The practical advantages of the direct Gooch process render the method worthy of serious consideration, especially in problems wherein suspended matter is an important factor.

None of the current methods for the determination of organic matter distinguish between stable and unstable organic matter. Only by resorting to one of the various methods for the determination of putrescibility can information be obtained regarding the putrescible and non-putrescible matter in partially purified sewage, and even under these conditions there is indicated only the relative predominance of putrescible and non-putrescible substances. It seems to be in this direction that the need is most urgent for methods applicable to the determination of the organic matter in crude and purified sewages.

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